

**RUTGERS**  
New Jersey Agricultural  
Experiment Station

Mass Spectrometry Facility  
Center for Advanced Food Technology  
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**CONFIDENTIAL**

**Mass Spectrometry Laboratory Analysis Report #7546**

**Flavor Analysis of Breyer's Natural Vanilla Ice Cream**

Dear Mr. Sheehan:

This is the report pertaining to the above-captioned samples that you submitted for flavor analysis.

**I Sample Log**

The following samples were received for analysis:

1. Breyer's Natural Vanilla Ice Cream, Production Code: 29230 A 07:58

**II Analysis Request**

The analysis request was to extract and analyze the flavors from the vanilla ice cream.

### **III Analysis Methodology**

The vanilla ice cream ( 10 g) was transferred to a borosilicate glass test tube sealed with Teflon-lined, screw cap closure, thawed, matrix-spiked with 10 µg of naphthalene-*d*<sub>8</sub> internal standard (1.0 ppm w/v) and mixed thoroughly using a lab vortexor. The thawed ice cream was then divided into 4 equal portions, transferred to glass vials and extracted with equal volumes (1:1) of methylene chloride. The layers were allowed to separate and then the methylene chloride extracts isolated and pooled together. The pooled extracts were centrifuged 30 minutes at 2500 rpm to clarify (separate any water or emulsion) then dried with anhydrous sodium sulfate. The dried extract was concentrated under a gentle stream of nitrogen to a final volume of approximately 0.5 mL then transferred to a Purge & Trap apparatus (Scientific Instrument Services, Solid Sample P&T system) and subjected to Purge & Trap-Thermal Desorption-GC-MS analysis as follows:

#### **Purge & Trap-Thermal Desorption-GC-MS**

Concentrated methylene chloride extract prepared as described above was evaporated to dryness in a stream of nitrogen gas inside the glass tubing of the purge & trap apparatus (SIS Solid Sample Purge & Trap Oven). Immediately upon reaching dryness the sample was subjected to P&T analysis by purging with nitrogen at 50 ml per minute for 30 minutes at 150°C. The exhaust of the P&T apparatus was fitted with a Tenax-TA adsorbent trap. The traps were then connected to the Short Path Thermal Desorption system and thermally desorbed directly into the GC-MS system for final analysis (SIS Model TD-4 Short Path Thermal Desorber). The thermal desorption conditions were 250°C for 5 minutes. A method blank was prepared and analyzed prior to the vanilla ice cream sample. Compounds detected in the method blank were disregarded in the data treatment of the test sample.

#### **GC-MS Analysis Methodology**

Analyses of Tenax traps prepared as described above were conducted using a Scientific Instrument Services (SIS) model TD4 Short Path Thermal Desorber interfaced to the Varian 3400 GC directly coupled to a Finnigan TSQ-7000 triple stage quadrupole tandem mass spectrometer equipped with an Xcaliber data

system. Thermal desorption conditions were 250°C for 5 minutes using sub-ambient, cryogenic GC column temperature programming. The GC was equipped with a 60 meter x 0.32 mm i.d. Guardian-ZB-5MS capillary column with a 1.0 µm film thickness (Phenomex). The mass spectrometer was operated in electron ionization mode (70 eV) scanning masses 35-350 once each second.

### **Materials**

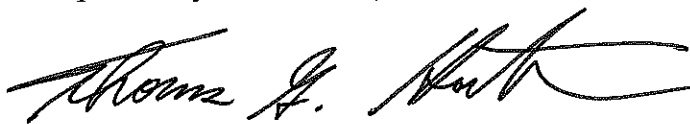
Naphthalene- $d_8$  used as internal standard for the study was purchased from Sigma-Aldrich Chemical Co, St. Louis MO. Methylene chloride was purchased from Thermo Fisher Scientific. All thermal desorption supplies were purchased from Scientific Instrument Services, Inc., Ringoes, NJ.

## **IV Results**

The GC-MS analysis data for the vanilla ice cream flavor is summarized in Table 1. The GC-MS chromatogram corresponding to the Table is presented in Figure 1. From left to right, the Table lists the MS scan number (from centroid of peak), peak area integration, peak identification and then concentration data expressed in parts per million (ppm w/v). The data is semi-quantitative and based on peak area ratio to the matrix-spiked internal standard (naphthalene- $d_8$ ) assuming a detector response factor of 1.0 with no correction for extraction efficiency.

If you have any questions or if I can be of further assistance to you then please don't hesitate to contact me.

Respectfully Submitted,

A handwritten signature in black ink, appearing to read 'Thomas G. Hartman', with a stylized flourish at the end.

Thomas G. Hartman, Ph.D.  
Mass Spectrometry Lab Director  
& Research Professor

## Attachments

- ▶ Table 1, Analysis Results Summary
- ▶ Figure 1, GC-MS Chromatogram
- ▶ Analysis Data Forms
- ▶ Photo of Test Sample

Table 1

Sheehan &amp; Associates, P.C., Project #7546

Breyer's Natural Vanilla Ice Cream

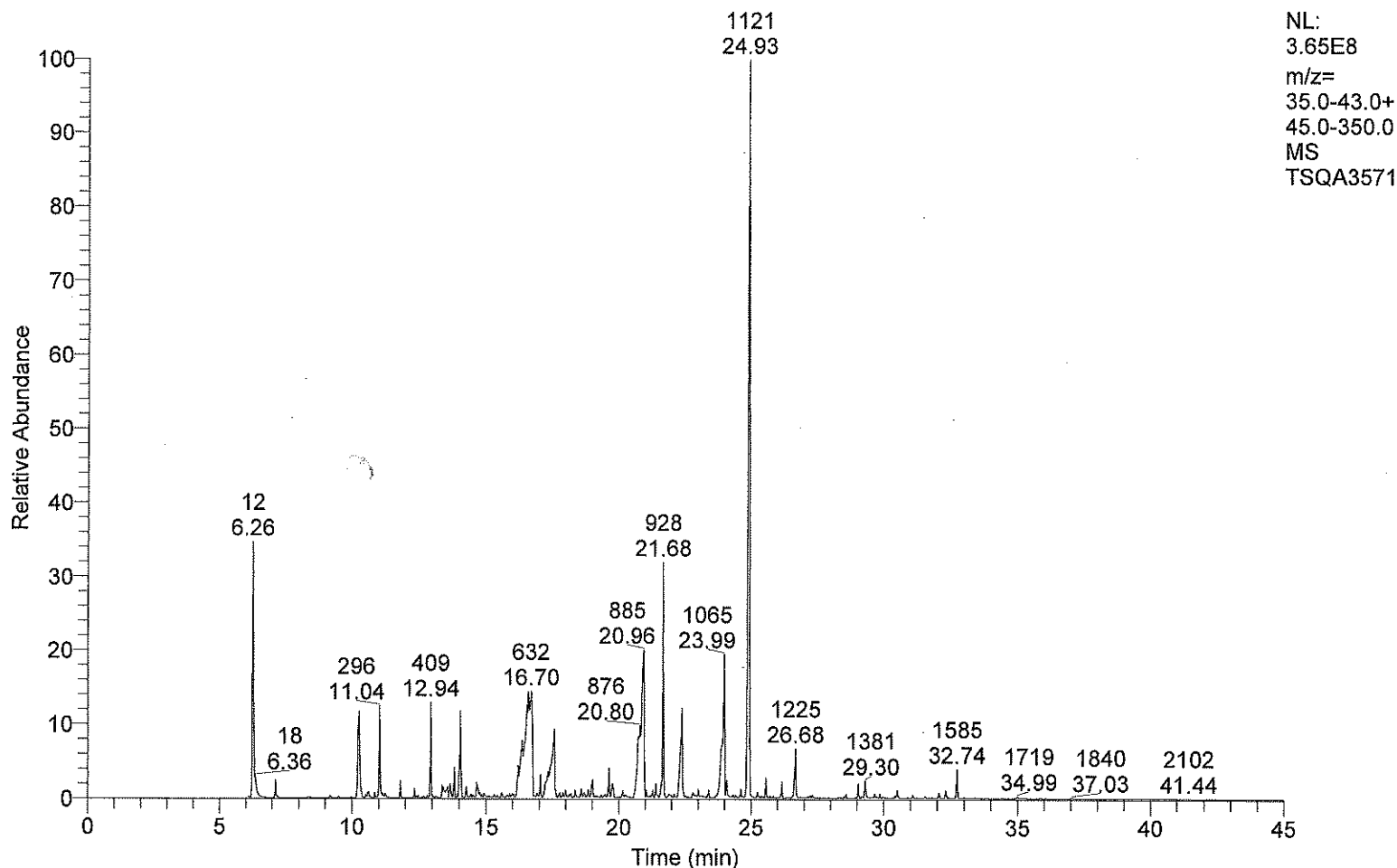
Production Code: 29230 A 07:58

Methylene Chloride Extract of 10.0 g with 1 ppm Matrix-Spiked Int. Std. by P&amp;T-TD-GC-MS

Data File = TSQA3571

MS	Area		Conc.
Scan #	Integration	Peak Assignment	PPM w/w
205	99298	acetic acid	0.022
250	3625596	diacetyl	0.797
342	268657	acetoin	0.059
447	440003	butyric acid	0.097
474	2288379	ethyl lactate	0.503
510	679180	dimethylsulfoxide (DMSO)	0.149
632	14688936	dimethyl sulfone	3.230
684	5573689	hexanoic acid	1.226
750	115042	benzyl alcohol	0.025
769	568416	heptanoic acid	0.125
806	540814	guaiacol	0.119
813	465017	nonanal	0.102
836	139435	maltol	0.031
885	12067851	octanoic acid	2.654
890	77124	benzoic acid	0.017
912	315936	2-methoxy-4-methylphenol	0.069
928	4547572	naphthalene-d8 (internal standard)	1.000
971	3843797	nonanoic acid	0.845
1007	187607	delta-nonolactone	0.041
1030	148583	2,4-decadienal	0.033
1065	7005324	decanoic acid	1.540
1121	25782638	vanillin	5.670
1140	125485	undecanoic acid	0.028
1158	376018	vanillyl ethyl ether	0.083
1195	335836	delta-decalactone	0.074
1225	1486537	lauric acid	0.327
1338	81955	gamma-dodecalactone	0.018
1365	268239	delta-dodecalactone	0.059
1381	405333	myristic acid	0.089
Total (excluding internal standard)			18.03

RT: 0.00 - 45.02



TSQA3571

Type: Unknown ID: 1 Row: 1

Sample Name: Breyer's Natural Vanilla Ice Cream (Production Code: 29230 A 07:58), DCM Extract, 150C/30min, matrix spiked with w/w 1.0ppm Int. Std. by P&T-TD-GC-MS

Study: Sheehan & Associates, P.C., LLN7546  
 Client: Mass Spectrometry - Dr. Tom Hartman  
 Laboratory:

Company:  
 Phone:  
 Instrument Method: C:\Xcalibur\methods\voc45solventdelay6min.meth  
 Processing Method:  
 Vial: 1  
 Injection Volume (µl): 10.00  
 Sample Weight: 0.00  
 Sample Volume (µl): 0.00  
 ISTD Amount: 0.00  
 Dil Factor: 1.00

# **SHORT PATH THERMAL DESORPTION DATA FORM** **MASS SPECTROMETRY LABORATORY**

ANALYST: Joe Scarsella PROJECT #: 7546 DATE: 11/7/19

PROJECT SPONSOR: Sheehan and Associates, P.C.

INSTRUMENT: SCIENTIFIC INSTRUMENT SERVICES INC., (SIS) MODEL TD-2  
 SHORT PATH THERMAL DESORPTION SYSTEM & ACCESSORIES

TYPE OF ANALYSIS: ☐ DIRECT THERMAL DESORPTION (DTD)  
☒ PURGE & TRAP - THERMAL DESORPTION (P&T-TD)

## **PURGE & TRAP AND DIRECT THERMAL DESORPTION CONDITIONS:**

SAMPLE DESCRIPTION: DCM extract of Breyer's Natural Vanilla ice cream

SAMPLE SIZE: \_\_\_\_\_ SAMPLE MATRIX: ☐ GAS ☐ LIQUID ☒ SOLID

PURGE & TRAP APPARATUS: ☒ SIS SOLID MATRIX SAMPLING OVEN

☐ SIS LIQUID PURGE VESSEL ☐ WHEATON LIQUID SAMPLER ☐ SKC PUMP

☐ CUSTOM APPARATUS: \_\_\_\_\_

PURGE GAS: ☒ NITROGEN ☐ HELIUM ☐ AIR PURGE GAS FLOW RATE: 50 mL/min

PURGE & TRAP TIME: 30 min TEMPERATURE: 150°C DRY PURGE TIME: 90 min

ADSORBENT TRAP: ☒ TENAX ☐ CARBOXEN-569 ☐ OTHER: \_\_\_\_\_

ADSORBENT TRAP BED VOLUME: ☐ 2cm ☐ 4cm ☒ 6cm ☐ 8cm

GLT DESORPTION TUBE INSIDE DIAMETER: ☐ 3mm i.d. ☒ 4mm i.d.

INTERNAL STANDARD: benzene-d<sub>6</sub>, toluene-d<sub>8</sub>, naphthalene-d<sub>8</sub> AMOUNT INTERNAL STANDARD: 1.0 ppm w/w

INTERNAL STANDARD SPIKE: ☒ MATRIX SPIKE ☐ ADSORBENT TRAP SPIKE

## **THERMAL DESORPTION CONDITIONS:**

DESORPTION TEMP.: 250°C DESORPTION TIME: 5 min INJ. TIME: 30 sec

INITIAL PURGE TIME: 10 sec TYPE OF ANALYSIS: ☐ GC ☒ GC-MS

COMMENTS: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

# TSQ-7000 MASS SPECTROMETRY ANALYSIS DATA FORM

## MASS SPECTROMETRY LABORATORY

DATA FILE(S): TSQA3571 PROJECT #: 7546 DATE: 11/7/19  
 PROJECT SPONSOR: Sheehan + Associates, P.C. ANALYST: Joe Scarsella  
 SAMPLE DESCRIPTION: Breyer's Natural Vanilla ice cream, Dcm extract

### GC CONDITIONS:

INSTRUMENT: Varian 3400 COLUMN: 2B5-ms SERIAL #: —  
 LENGTH: 60m DIAMETER: 0.32mm FILM THICKNESS: 0.25um  
 TYPE OF INJECTION: ☒ SPLIT ☐ SPLITLESS ☐ ON-COLUMN  
☐ HEADSPACE ☒ THERMAL DESORPTION  
 INJ. LINER: ☐ CYCLOID ☐ 2mm Direct ☒ 4mm Direct ☐ (Other) —  
 INJECTION VOLUME: — INJECTOR TEMPERATURE: 250°C  
 SEPTUM PURGE: ☒ YES ☐ NO SPLIT RATIO: 10:1  
 CARRIER GAS: He CARRIER FLOW (ml/min.): — or PSI HEAD PRESS.: 20psi  
 TEMP. PROGRAM: -20°C → 260°C @ 10°C/min  
 GC-MS INTERFACE LINE TEMPERATURE: 280°C GC MAINTENANCE:  
☒ SEPTUM CHANGE ☐ CLEAN & SILANIZE INJ. LINER ☒ Column Bakeout

### MASS SPECTROMETER CONDITIONS:

INSTRUMENT: Finnigan MAT TSQ7000 DATA SYSTEM: Xcaliber  
 IONIZATION MODE: ☒ EI (70 eV) ☐ CI ☒ +ION ☐ -ION ☐ CID (MS-MS)  
 IF CI THEN INDICATE REAGENT GAS: ☐ METHANE ☐ ISOBUTANE ☐ AMMONIA  
 ION SOURCE TEMP.: 185°C FILAMENT EMISSION CURRENT: 400uA  
 TYPE OF ANALYSIS: ☒ GC-MS ☐ DCI ☐ EI/CI DIRECT PROBE ☐ BATCH INLET  
☐ SIM MODE: ION(S) MONITORED: —  
☐ MS-MS MODE: CID PRESSURE mT: — COLLISION ENERGY V: —  
☐ SRM MODE: DAUGHTER ION(S) MONITORED: —  
 DCI OR DIRECT PROBE TEMPERATURE RAMP: —  
 MASS RANGE: 35-350 SCAN RATE (seconds/decade): 1.0 sec  
 TUNE FILE: EI-Tune METHOD FILE: VOC45 solvent delay 6 min. meth  
 COMMENTS: —